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Exploring the potential of calcined montmorillonite-kaolinite clay with medium percentage of clay minerals for low-carbon cements

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Abstract. Expanding the raw material base and diversifying supplementary cementitious materials based on distribution, availability, and competitiveness with traditional materials is a key focus for promoting the production and application of low-carbon cements. This approach is crucial for ensuring the sustainable development of the cement industry. Extensive research and practical experience in recent decades have highlighted the promising potential of using thermally activated clays to create low-emission cements. Calcined clays have now become significant and growing part of the range of reactive aluminosilicates suitable for use as primary or supplementary sources in producing clinker-free or low-clinker cements. This is facilitated by the abundant reserves of clay raw materials and the high reactivity of activated clays. The practical implementation of thermally activated clays followed extensive, long-term studies on the potential use of various types of clays. Research focused on the influence of chemical and mineralogical compositions, activation methods, and other factors on their reactivity. While important parameters for kaolin clays, such as kaolin content, optimal temperature and duration of thermal treatment, and fineness of grinding, are well-defined and included in standards, these aspects are not thoroughly studied for polymineral and montmorillonite clays. This study examines the impact of temperature and duration of calcination on the reactivity of montmorillonite-kaolinitic clay with an average clay minerals content and explores its potential in environmentally-oriented low clinker cements.

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1. Introduction

The development of modern Portland cements (PC) that meet the requirements of sustainable development has led to the need to expand and adapt the mineral raw material base of the cement industry for the production of eco-friendly binders. In recent decades, research aimed at increasing the variety of supplementary cementitious materials (SCMs) for composite and multicomposite cements has intensified [1–7]. A reasonable approach to expanding the raw material diversity of SCMs is to search for more widespread natural and technogenic resources that are widespread and readily available compared to traditional pozzolans additives and mineral fillers. The growing demand for affordable and high-quality SCMs has sparked increased interest in carbonate and clayey raw materials as both individual and binary SCMs to PC [1, 8–10]. As a result, at the current stage of PC development, these rocks are not only practical non-alternative mineral sources for the production of PC clinker as a basic part of PC but also SCMs, among other natural and man-made resources, adapting cements to the current environmental requirements for reducing the impact of cement production on the environment.

Clay rocks, which account for 70 % of sedimentary rocks, are known for their large reserves, widespread deposits around the world, diverse chemical and mineralogical compositions, and varied content and structure of main rock-forming minerals. Among the many clay minerals, kaolinite and smectite are considered to have the highest value when used in the activated state as SCMs for various binder systems. Kaolin clays, particularly metakaolin, have been extensively studied for their application as SCMs and are used in the production of cement and concrete on an industrial scale. However, the global reserves of kaolin clays are limited compared to other types of clays, and they are predominantly found in regions with tropical and subtropical climates. In regions with arid and cold climates, 2:1 clay minerals, such as smectite, illite, chlorite, and mixed-layer minerals more common [9]. This has led to a significant amount of research focusing on clays with medium and low kaolin content, non-kaolin clays, and polymineral clays to identify alternative sources of clay raw materials for the production of reactive aluminosilicates [11–20].

A number of studies [20–32] have established the effectiveness of smectites in initial, modified, and activated states when used as SCMs for PC. The positive effect on the physical and mechanical properties of cement composites depends on the chemical and mineralogical compositions, crystallochemical features of minerals, method and mode of activation, dispersity, concentration of montmorillonite additives, and other factors. Thus, Vallina et al. [19], reported that active smectites consisted in starting form an amorphous content of approximately 80 wt% exhibited pozzolanic activity comparable to clay with 30–35 % kaolinite as determined by the R3 test. According to Liu [32], the addition of 8–10 % Na-bentonite with an average particle size of 10 μm resulted in 77.5 % increase in the compressive strength (CS) and a 54.5 % increase in flexural strength of cement-sand mortars. The strengthening effect of Ca- and Mg-bentonites was found to be less significant. The permeability reduction was highest with Mg-bentonite addition (137.3 %), followed by Na- (115.7 %) and Ca-bentonite (101.9 %).

If we consider kaolin clays, aspects, such as the limit of kaolin content, optimal temperature and duration of heat treatment, fineness of grinding, etc., are determined by the results of complex studies and included in the standards [33]. However, for montmorillonite clays these issues have not been fully studied. An analysis of the results of studies of the influence of calcined bentonite on the physical and mechanical properties of cement composites shows that not all results are consistent in terms of positive influence or level of improvement of indicators. This inconsistency is likely due to various parameters, such as chemical-mineralogical composition of clays, particle size, purity, crystallinity of clays, temperature and time of calcination, which need to be considered and systematized. The optimal calcination temperature for montmorillonite clays reported in the literature were 600 °C [20], 750 °C [21], 800 °C [19, 22–25], 830 °C [26–28], 850 °C [29], 900 °C [30, 31]. Additionally, only a few papers provide the exact quantitative mineral composition of clays [19, 24, 31, 34, 35]. The optimal duration of heat treatment according to study results can range be from 1 to 5 h. Determining the optimal heat treatment modes has its own peculiarities for clays with a mixed mineralogical composition.

In the present study, we investigate the influence of temperature and calcination duration on the reactivity of montmorillonite-kaolin clay, as well as its impact on the properties of fresh and hardened cement pastes.

2. Methods and Materials

The ordinary PC and montmorillonite-kaolinite clay were used as starting materials in this study. The chemical compositions of the mineral sources are listed in Table 1. The mineralogical composition of PC was as follows: C_3S – 72.0 %, C_2S – 9.8 %, C_3A – 5.0 %, C_4AF – 11.4 %. The mineralogical composition of montmorillonite-kaolinite clay from the Biklyansky deposit of Russia (% wt.) was:

montmorillonite (PDF 03-0015) $(\text{Na,Ca})_{0.3}(\text{Al,Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot \text{H}_2\text{O}$ – 36 %;

kaolinite (PDF 00-058-2028) $(\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4)$ – 13 %;

quartz (PDF 01-079-1910) (SiO_2) – 22 %;

albite (PDF 01-084-0752) $(\text{NaAlSi}_3\text{O}_8)$ – 7 %;

microcline (PDF 01-084-1455) $((\text{K}_{0.95}\text{Na}_{0.05}) \cdot \text{AlSi}_3\text{O}_8)$ – 8 %;

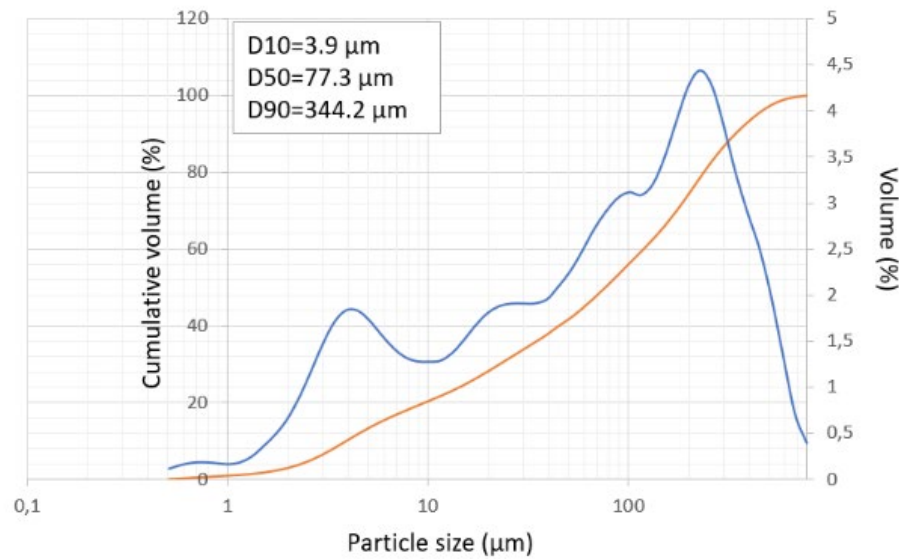
muscovite (PDF 01-070-1869) $(\text{K}_{0.77}\text{Al}_{1.93}(\text{Al}_{0.5}\text{Si}_{3.5})\text{O}_{10} \cdot (\text{OH})_2)$ – 10 %;

clinochlore (PDF 01-070-1869) $((\text{Mg}_{2.96}\text{Fe}_{1.55}\text{Fe}_{0.136}\text{Al}_{1.275}) \cdot (\text{Si}_{2.622}\text{Al}_{0.376}\text{O}_{10}) \cdot (\text{OH})_8)$ – 4 %.

Table 1. Chemical composition of the starting materials (% wt).

Material	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO
PC	20.7	4.5	3.5	65.0	3.1
Clay	50.9	20.73	7.6	0.84	2.09

The chemical composition of the source materials was determined using an atomic emission spectrometer (OPTIMA 4300 DV). The clay was ground using a ball mill. The particle size distributions of the source materials were measured using a laser particle size analyzer (Horiba La-950V2). The clay was dispersed in ethanol using ultrasound as the dispersion medium. Details of the size distributions are provided in Fig. 1. The ground calcined clay (CC) was calcined at 700, 800, and 900 °C with a heating rate of 3.3 °C/min. Dwell times of 1 and 2 h were used.

**Figure 1. Particle size distribution of the CC.**

X-ray diffraction (XRD) and thermal analyses (TG/DSC) were conducted on ground CC. The XRD results were obtained using a D2 Phaser X-ray diffractometer in a Bragg-Brentano θ - 2θ configuration with Cu K α radiation operating at 40 kV and 30 mA. Data analysis was performed using the DIFFRAC plus Evaluation Package EVA Search/Match and PDF-2 ICDD database. The mineralogical composition of the clays was determined by analyzing the X-ray diffractograms of the software product Diffrac.eva V3.2. A STA 443 F3 Jupiter simultaneous thermal analysis apparatus was used for the TG/DGA. The CC was heated from 30 to 1000 °C at a heating rate of 10 °C/min. The data were analyzed using Netzsch Proteus Thermal Analysis software. Scanning electron microscopy (SEM; FEI XL-30ESEM) was performed at an accelerating voltage of 20 keV. FTIR spectra were recorded using a Spectrum 65 (Perkin-Elmer) ranging from 4000 to 600 cm⁻¹.

The PC and calcined clay were mixed for 3 minutes using a mixer. Water was then added to the dry mixture, and mixing continued for 5 minutes and then the pastes was poured into 20 × 20 × 20 mm cubic steel molds and compacted on a vibration table to remove trapped air. Simultaneously, all the specimens were covered with a plastic sheet to prevent moisture evaporation. After 24 hours of casting, the specimens were de-molded. They were then cured in water at 20 ± 1 °C until the testing age. Two sets of samples were prepared. The CS of the first set was tested after 2, 7, 28 and 90 days of curing at ambient temperature (25 °C) and 98 % relative humidity. The second set of hardened cement pastes was tested after steam curing, following a thermal curing program of 3 h to reach the desired temperature, 6 h of dwell time at 80 °C, and 4 h of cooling. Mechanical tests were conducted by applying a vertical load between the two parallel surfaces during casting. Each CS determination was based on the average of six measurements from the same cast. Water adsorption was determined by calculating the ratio of the weight increase of samples after immersion in water to their dry weights (the samples were dried to a constant mass at a temperature of 102–105 °C). Mortar samples were prepared in the same order, with a PC:sand ratio of 1:3, a water/PC(PC+CC) ratio of 0.5, and sample sizes of 4 × 4 × 16 cm.

3. Results and Discussion

3.1. Characterization of As-received and Calcined Clay

In order to determine the optimal calcination mode, including temperature and duration, studies were conducted on the influence of calcination temperature at 700, 800 and 900 °C and duration for 1 and 2 h on the mineralogical and phase compositions. Fig. 2 shows the X-ray diffractograms of the as-received clay and clays calcined at 700, 800, and 900 °C for 1 h. The thermal treatment of clay resulted in an increase in the loss of crystallinity with an increase in calcination temperature, which is related to the structural transformations of kaolinite, montmorillonite, and muscovite. The calcination of clay at 700 °C resulted in decomposition to an amorphous phase and the disappearance of the peak assigned to kaolinite while the reflection assigned to montmorillonite was reduced in intensity, indicating a decrease in crystalline montmorillonite content. The montmorillonite transformed into an anhydrous form Na-Mg-Al-Si₄O₁₀ (PDF 07-0304). Further increase in calcination temperature to 800 °C led to an increase in amorphous phase content, and the percentage of crystalline montmorillonite decreased from 43 to 27 %. These observations are consistent with existing literature, indicating that dehydroxylation of 2:1 clay minerals disrupts the stacking of layers in the crystallographic c-axis but does not disrupt along the a and b-axes within the layer plane [36, 37]. The different temperature of dehydroxylation for kaolinite and montmorillonite is due to water molecules being easier to remove from the kaolinite structure compared to montmorillonite [38]. Since calcined products have structural differences, the solubility of Si and Al of metakaolin in alkaline conditions is greater than that for metamontmorillonite by 4 and 12, respectively [37]. This means that considering the presence of both kaolinite and montmorillonite, clay after thermal treatment contains reactive Si and Al, which have different chemical reactivity, and the content of highly reactive components is proportional to the percentage of kaolinite in the clay. Further increase in treatment temperature up to 900 °C results in the disappearance of montmorillonite reflecting its amorphization, and weakening of peaks associated with muscovite.

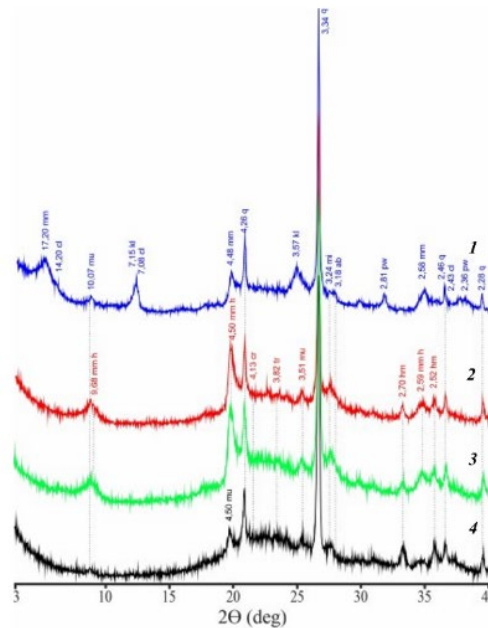


Figure 2. X-ray diffractograms of as-received clay (1), clay calcined at 700 °C for 1 h (2), clay calcined at 800 °C for 1 h (3), clay calcined at 900 °C for 1 h (4)

(Mm – montmorillonite, Q – quartz, Ca – calcite, Ab – albite,

Mi – microcline, Tr – tridymite, Cr – cristobalite, Mu – muscovite, Pw – pseudowollastonite, Cl – chlorite, Ki – kaolinite, Hm – hematite).

The increase in the duration of calcination from 1 to 2 h did not reveal any significant differences as shown in Fig. 3. This allows us to conclude that 1 h is sufficient for the thermal activation of clay. Therefore, clay samples obtained after calcination for 1 h were used in further research.

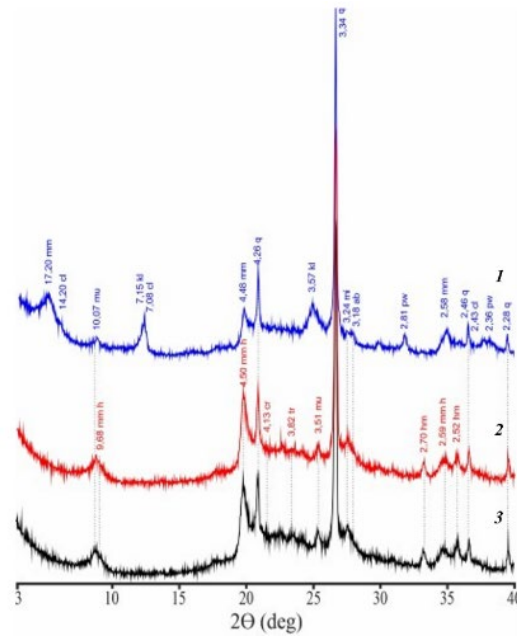


Figure 3. X-ray diffractograms of as-received clay (1), clay calcined at 800 °C for 1 h (2), clay calcined at 800 °C for 2 h (3) (Mm – montmorillonite, Q – quartz, Ca – calcite, Ab – albite, Mi – microcline, Tr – tridymite, Cr – cristobalite, Mu – muscovite, Pw – pseudowollastonite, Cl – chlorite, KI – kaolinite, Hm – hematite).

XRD results are confirmed and complemented by thermal analysis data presented in Figs. 4, 5.

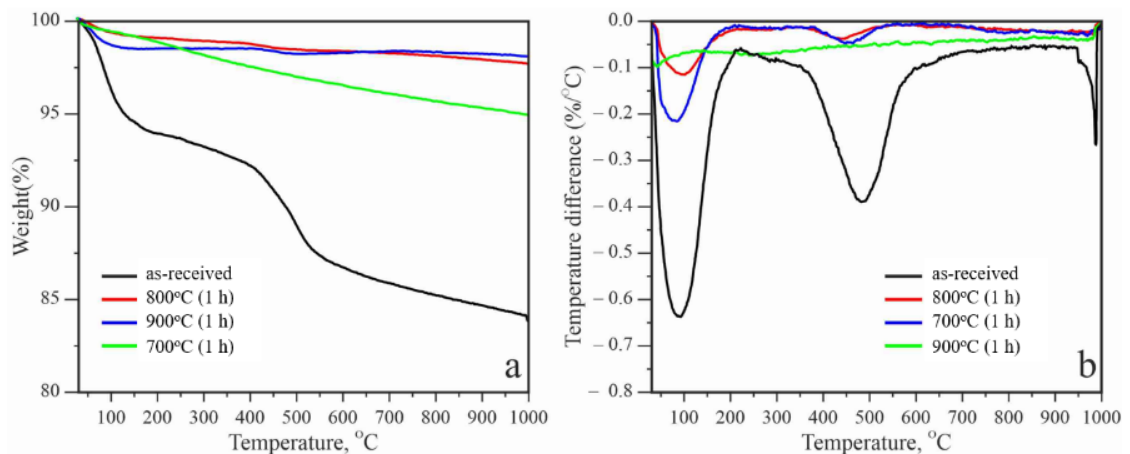


Figure 4. DTA and TG curves for as-received and clays calcined at 700, 800, and 900 °C for 1 h.

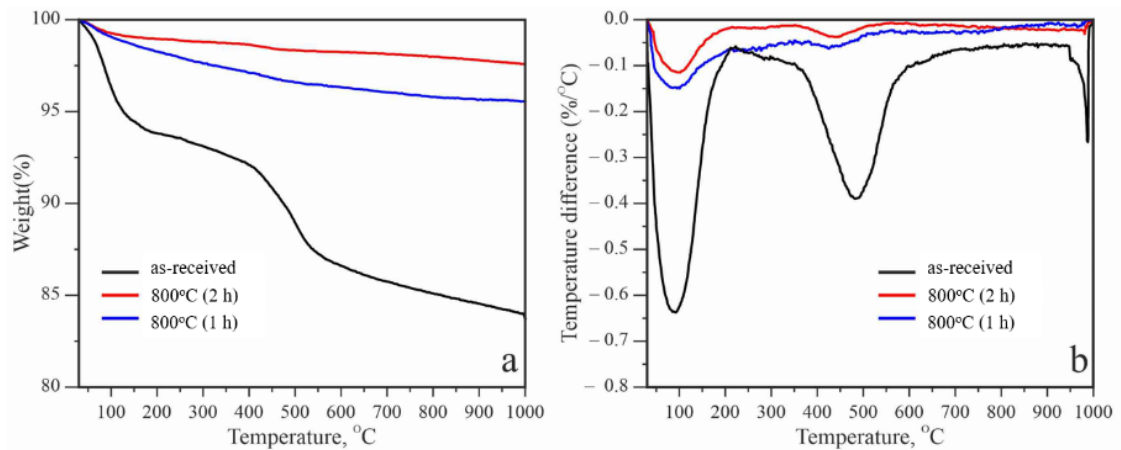


Figure 5. DTA and TG curves for as-received and clays calcined at 800 °C for 1 and 2 hours.

The DTA curves of as-received clay and clays calcined at 700 and 800 °C show 2 endothermic peaks accompanied by the highest mass losses in their respective temperature ranges. The first DTG peak position corresponds to the dehydration phase (in the region of 30–140 °C) – the loss of adsorbed and interlayered water was registered at 91.4 °C, and the second peak is related to the dehydroxylation stage (in the region of 425–800 °C) – the loss of OH groups from the structure of montmorillonite at 483.3 °C. It can be observed that the intensity of both peaks becomes weaker with increasing temperature and duration of calcination, and they eventually disappear after treatment at 900 °C. It should be noted that for raw clay, the mass loss in the range of 425–700 °C was 5.73 %, 700–800 °C was 0.64 %, 800–900 °C was 0.56 %, and 900–1000 °C was 0.79 %. This may indicate a decrease in the intensity of reactions in the dehydroxylation and amorphization stages, while an increase in mass loss in the region of 900–1000 °C is characteristic of muscovite decomposition.

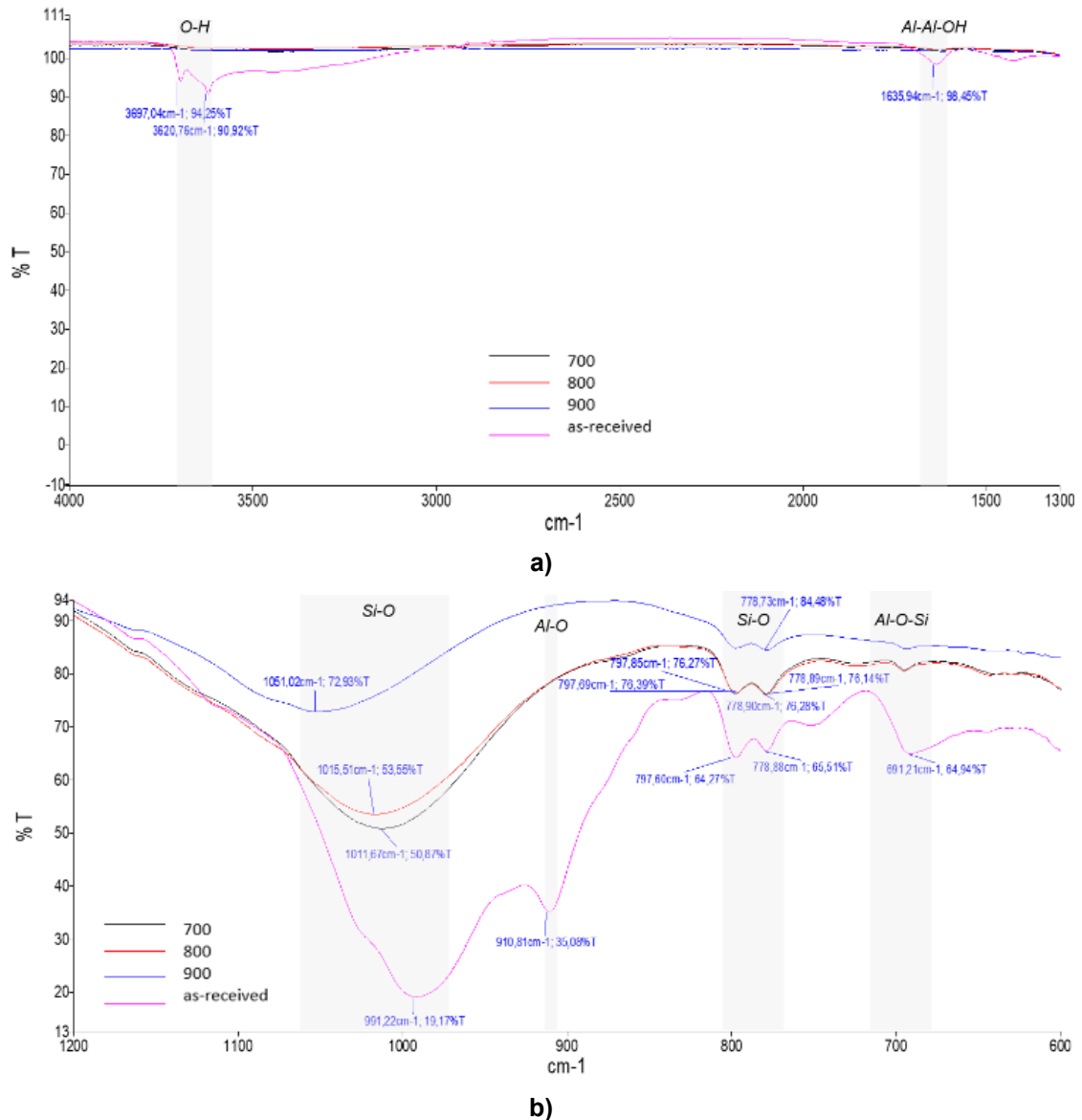


Figure 6. FTIR spectra of as-received and calcined clays: a) – 1300–4000 cm^{-1} , b) – 600–1200 cm^{-1} .

Fig. 6 shows the FTIR spectra of as-received and calcined clays. The stretching bands [40] (2800–3800 cm^{-1}) assigned to the structural hydroxyl and adsorbed water in clay minerals disappear at a treatment temperature 700 °C. The vibrations assigned to the aluminosilicates in the region 400–1200 cm^{-1} shift to higher wavenumbers with an increase in thermal treatment temperature [39, 40]. The position assigned to Si-O at 991 cm^{-1} for the as-received clay shifted to 1011, 1015, and 1051 cm^{-1} for clays calcined at 700, 800, and 900 °C, respectively. This shift is attributed to increased structural disorder of local bonding environments within the tetrahedral sheet of montmorillonite [41]. Bands corresponding to Al-R(Al, Mg, Fe)-OH vibrations (where R is an octahedral site of Al, Mg or Fe) at 910 cm^{-1} are not detected in calcined clays, justifying the structural disorder in the octahedral sheet [42]. With increasing calcination temperature, the Si-O stretching bands around 1030 cm^{-1} broaden, indicating the presence of amorphous silica [40]. The decrease in the intensity of (H–O–H) bending vibrations of water molecules at 1635 cm^{-1}

and (Al–Al–OH) of octahedral Al in montmorillonite at 916 cm^{-1} also indicates the decomposition of the smectite mineral structure [43, 44]. The stretching bands at 691 cm^{-1} assigned to Al–O–Si become weaker, indicating the rupture of the octahedral and tetrahedral sheets, and hence the disruption of long-range periodicity along the c-axis [45,46].

The results of the evaluation on the influence of temperature and duration of thermal treatment on the reactivity of CC, as estimated by the CS of hardened cement paste with 15 % CC additions, are presented in Fig. 7. The data shows that calcination at $800\text{ }^{\circ}\text{C}$ for 1 h provides the highest reactivity compared to other calcination methods. These findings are consistent with previous studies [19, 22–25], which indicated that the peak reactivity occurs at a calcination temperature of around $800\text{ }^{\circ}\text{C}$. Additionally, in this study, the clay containing metamontmorillonite used exhibited slightly higher reactivity with a 37 % amorphization after calcination compared to the clay containing fully amorphized montmorillonite after calcination. These results align with Vallina et al.'s findings [19], suggesting that the optimal calcination temperature should not completely amorphize the montmorillonite phase and does not necessarily coincide with the maximum amorphization. In the authors opinion, the reason for this is an intermediate situation between the hydroxylated Q3 of montmorillonite and condensed Q4 species, as well as the presence of Al(V), which may justify the highest reactivity. Conversely, a decrease in clay reactivity was observed after calcination at $900\text{ }^{\circ}\text{C}$, resulting in the total amorphization of montmorillonite. This decline could be attributed to the reduction in the specific surface area of the clay after treatment at this temperature, as was established through visual observations and requires further research for a quantitative assessment of these changes. Subsequently, the clay obtained by thermal activation at $800\text{ }^{\circ}\text{C}$ for 1 hour was utilized in further studies.

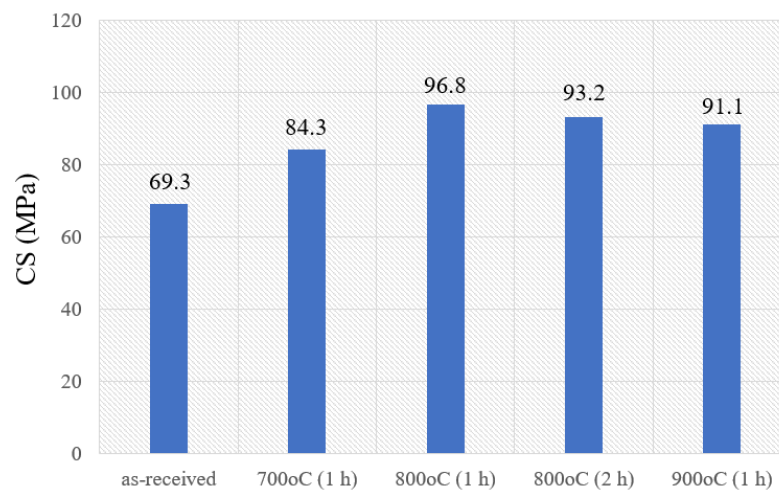


Figure 7. The effect of temperature and duration of calcination on the CS of hardened cement paste after steam curing, incorporated with 15 % of CC.

3.2. *Effect of Calcined Clay on the Properties of the Fresh and Hardened Pastes*

Figs. 7, 8 show the results of experiments evaluating the influence of CC on the standard consistency, CS, density, and water absorption of hardened cement pastes. According to the data presented in Fig. 8 increasing the CC content from 0 to 20 % leads to increased water demand from 27.5 to 29.4 %. As a result, hardened cement pastes incorporated with CC demonstrated lower density and higher water adsorption when compared to reference samples. Moreover, as shown in Fig. 9, increasing the loading of CC up to 20 % deteriorates mechanical characteristics both during curing under normal conditions and after steam curing. However, the level of strength reduction depended on the additive content and curing time. Thus, the greatest strength reduction of specimens at the introduction of CC was observed at the age of 2 days – 10.1–27.2 %. At further curing, the specimens with the content of 5–10 % additives at the age of 28 and 90 days were inferior to those without additives by 3.0–6.5 % in strength after curing by 3.0–6.5 %, and by 1.8–2.3 % after curing under normal conditions. The introduction of CC in amounts exceeding 15–20 % resulted in a decrease in the CS of hardened cement paste by 14.7–31.1 % depending on the curing time. The results indicate that with dosages – up to 10 % thermally activated montmorillonite-kaolin clay, the reactive aluminosilicate phase shows activity, evident from 7 days of curing. This activity is sufficient for replacing PC without significantly reducing hardened cement paste CS at 7, 28, and 90 days and after steam curing. Increasing the replacing content beyond 10 % causes a significant decrease in the CS of hardened cement paste due to dilution of PC with less reactive CC with a relatively low specific surface area.

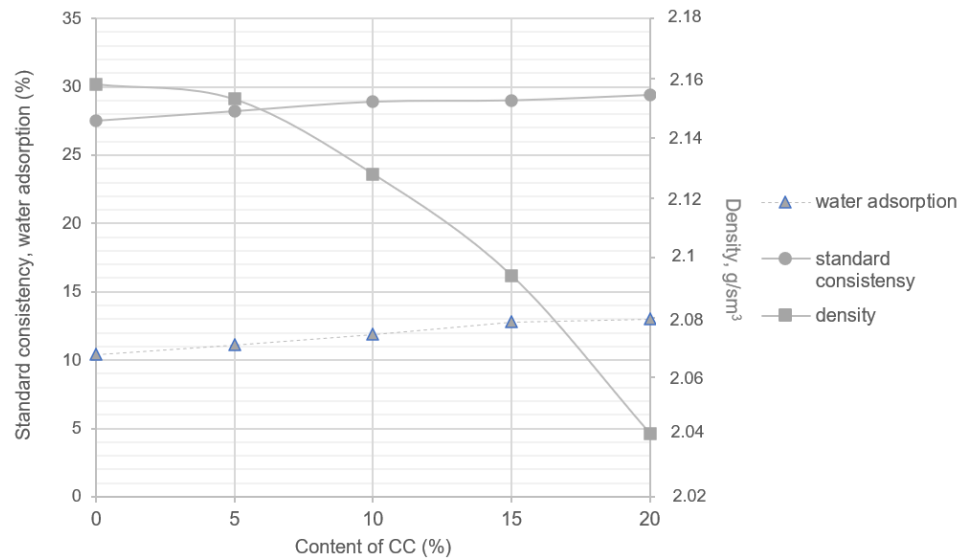


Figure 8. Standard consistency, water absorption, and density of cement pastes, incorporated with 0–20 % of CC.

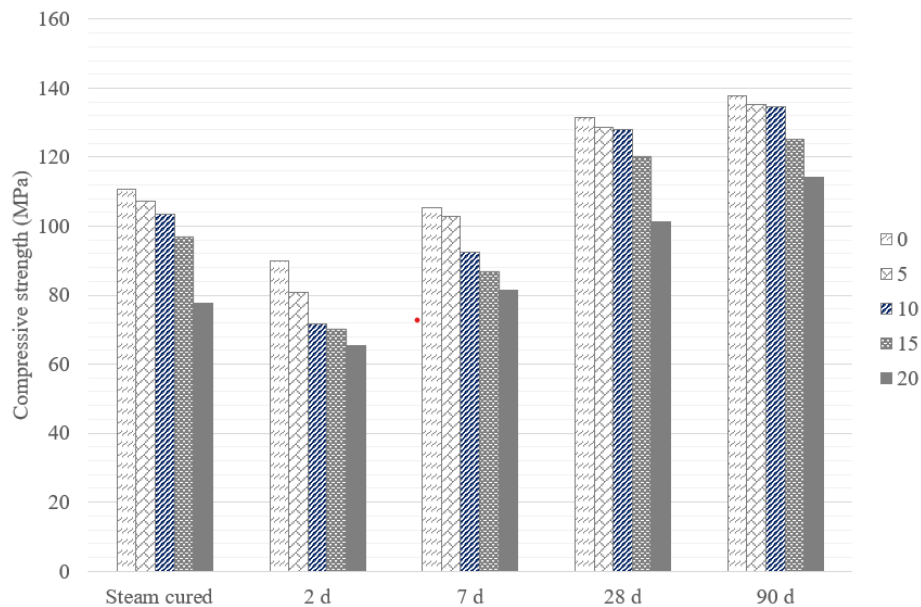


Figure 9. CS of the hardened cement pastes, incorporated with 0–20 % of CC.

3.3. Effect of Calcined Clay on the Mechanical Properties of the Hardened Mortars

Table 2 shows the results of experiments evaluating the influence of CC on CS and flexural strength (FS) of hardened mortars.

Table 2. Mechanical properties of hardened mortars.

CC (%)	CS (MPa)			FS (MPa)		
	2 d	7 d	28 d	2 d	7 d	28 d
–	30.0	48.3	54.9	7.0	7.1	8.6
15	28.1	45.9	52.0	6.5	6.7	8.0

As can be seen from the presented data, mortar samples with a 15 % activated clay additive are only slightly inferior in strength to control samples.

4. Conclusions

Thermally activated clays, with their abundant raw material base and high reactivity, show promise as aluminosilicate materials for producing low- and no-clinker cements. However, incorporating clays with

their wide range of chemical and mineralogical compositions to blended cements requires comprehensive studies. These studies must consider all factors that impact effectiveness, quantitative criteria for assessing suitability and potential, and determine the best way to utilize these materials.

The results presented in this study show the optimal conditions for thermal activation of mixed montmorillonite-kaolinite clay with the montmorillonite content of 36 % and kaolinite content of 13 % are 800 °C for 1 h. The maximum activity of the clay as observed in the study resulted in a 37 % decrease in the crystallinity of montmorillonite. It was found that thermally activated clay with this mineral composition, with a d₅₀ of 77.3 µm, can be used as a SCM to PC in amounts of 5–10 % without a significantly reducing the CS of hardened cement paste at 7, 28 and 90 days, as well as after steam curing. Furthermore, it is possible to enhance the efficiency of using this CC use as SCM to PC by adjusting the particle size and incorporating water-reducers. Therefore, further research in these areas is recommended to explore the potential for increasing PC replacement and improving physical and mechanical properties of blended cements.

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